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TECHNICAL REPORT

TEST REPORT

T61-8-1

COLORIMETRIC DETERMINATION OF METALLIC TIN

IN PROPELLANTS BY USE OF HEMATOXYLIN

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March 1961



FRANKFORD ARSENAL

338000

PHILADELPHIA 37, PA

Frankford Arsenal
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Test Report T61-8-1
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COLORIMETRIC DETERMINATION OF METALLIC TIN
IN PROPELLANTS BY USE OF HEMATOXYLIN

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I. INTRODUCTION

Metallic tin is frequently added to propellants to act as a flash reducer (1). The determination of metallic tin in propellants has been performed iodometrically or polarographically, after adding sulfuric and nitric acids, evaporating to fumes of sulfuric acid, and treating again with nitric acid (6). The determination has also been performed gravimetrically, after treatment with nitric acid, filtration, and ignition to tin dioxide (6). It is sometimes necessary to correct for impurities in the tin dioxide by volatilizing the tin with ammonium iodide (2, 6). When the tin is present in the propellant as tin dioxide, the determination must be performed gravimetrically (6), since tin dioxide is not soluble in ordinary solvents.

Inasmuch as the above methods are somewhat tedious, this laboratory undertook the development of a rapid colorimetric method. Hematoxylin was selected as the reagent, since it seemed to be one of the few reagents for the colorimetric determination of tin that was applicable in the presence of lead, an element that is frequently found in propellants.

The use of hematoxylin for the colorimetric determination of tin seems to have been first proposed by Tartakovskii (7), who made the solution slightly alkaline with sodium hydroxide, added hematoxylin, and compared the red color with standards. A solution of stannous tin was used but some oxidation probably took place during the color development. The method is unreliable because hematoxylin itself produces a red color above a pH of 8 (for this reason hematoxylin finds some application as an indicator (9)). Charlot (3) investigated the use of hematoxylin as a qualitative reagent for tin and other metals. Teicher and Gordon (8) developed the color at pH 0.8, but the pink color produced was not too sensitive and did not follow Beer's law. Kojima (5), using oxidized hematoxylin, developed the color in a solution containing 2 drops of sulfuric acid (10%) per 5 ml volume.

Since none of the above hematoxylin methods for the determination of tin was considered entirely adequate, it was deemed advisable to investigate the factors affecting the colorimetric determination of tin by hematoxylin.

II. SUMMARY

A method is proposed for the colorimetric determination of metallic tin in propellants by the use of hematoxylin. A 0.3 gram sample is treated with a mixture of nitric, sulfuric, and perchloric acids, and the solution is evaporated to fumes of sulfuric acid. An aliquot is taken, hematoxylin, gelatin, and sodium acetate are added, and the solution is heated for 15 minutes in boiling water. The factors influencing the color were studied, including the effect of amount of gelatin solution, amount and age of hematoxylin solution, initial acidity, amount of sodium acetate solution (pH), volume at which maximum intensity of the color is developed, order of addition of reagents, time interval between the addition of hematoxylin and sodium acetate, heating time in boiling water, temperature at which the color is read, stability of the color, valence of tin, wave length at which the color is read, and interferences. The method of developing the hematoxylin color is probably applicable to the determination of other metals as well as tin.

III. RECOMMENDATIONS

It is recommended that the method described in this report be incorporated into MIL-STD-286 and other applicable government specifications.

IV. STUDY

A. STUDY OF FACTORS AFFECTING THE COLORIMETRIC DETERMINATION OF TIN BY HEMATOXYLIN

Effect of Amount of Gelatin Solution

The lake precipitated unless gelatin was present. The following experiment was performed to find out how much gelatin was desirable. Five milliliter aliquots of Standard Tin Solution No. 2 (0.060 mg tin and 0.2 ml sulfuric acid) (see Reagents) were pipetted into 50 ml volumetric flasks. Fifteen milliliters of water, 5 ml of hematoxylin solution (0.1%), various amounts of gelatin solution, and 10 ml of sodium acetate solution (50%) were added. The solutions were heated in a boiling water bath for 13 minutes, cooled, and diluted to the mark. The transmittance was then measured at 585 millimicrons with a Beckman Model B spectrophotometer that had been set to 100 per cent transmittance with water. The results obtained (Figure 1) showed that the amount of gelatin was not too critical. The use of 4 ml is recommended.

Effect of Amount of Hematoxylin Solution

The effect of the amount of hematoxylin solution was investigated, using the same type of experiment as for gelatin. The amount of hematoxylin was somewhat critical (Figure 2). Four milliliters of the reagent, measured accurately, is recommended. The use of a hematoxylin solution in 95% ethyl alcohol containing 1 drop of hydrochloric acid per 100 ml, as recommended by Teicher and Gordon (8), is essential. Without the hydrochloric acid the solution darkened rapidly and erratic results were obtained. It is recommended that the hematoxylin reagent be prepared fresh daily. The blanks tended to increase as the hematoxylin solution aged from 10 hours to 1 week; however, during the same period the intensity of the color obtained for the tin increased somewhat (even after setting the spectrophotometer at 100 per cent transmittance with the blank). The blanks leveled off as the hematoxylin solution aged from 1 week to 2 weeks, but at the same time the intensity of the tin color decreased somewhat. The hematoxylin solution can be used for up to 8 days, if the curve is prepared on the same day the sample is analyzed, or if a standard is run at the same time as the sample. It was not necessary to filter the hematoxylin reagent, since a clear solution was always obtained.

Effect of Initial Acidity

Ten milliliters of sulfuric acid was the optimum amount that should be present in the 250 ml volume from which the 5 ml aliquot was taken for the color development. The presence of more sulfuric acid than 10 ml made it difficult to buffer the solution with sodium acetate; the presence of less sulfuric acid can cause hydrolysis of the tin. The normality of the solution in the 250 ml flask was about 1.4; the normality of the solution after taking the 5 ml aliquot and adding water, hematoxylin, and gelatin was about 0.26.

Effect of Amount of Sodium Acetate Solution (pH)

The color intensity and pH increased with increasing amounts of sodium acetate solution (50%), but leveled off at about 10 ml (Figures 3 and 4). The intensity of the yellow color of the blank increased with increasing amounts of sodium acetate (Figure 5). From a consideration of Figures 3, 4, and 5, the choice of 10 ml of sodium acetate solution (50%) (which gave a pH of 5.4) seemed satisfactory. Better results were obtained when the sodium acetate solution was added at one stroke from a graduate, rather from a pipet.

Effect of Volume at Which the Color is Developed

It is recommended that 15 ml of water be added to the 5 ml aliquot, so that the volume is about 38 ml when the solution is heated. When the water was omitted low results were obtained.

Effect of Order of Adding Reagents

Satisfactory results were obtained when the reagents were added in the following order: hematoxylin, gelatin, sodium acetate; or hematoxylin, sodium acetate, gelatin; or gelatin, hematoxylin, sodium acetate. Some of the lake precipitated when the reagents were added in the following order; sodium acetate, hematoxylin, gelatin; or gelatin, sodium acetate, hematoxylin. Apparently the sodium acetate must not be added before the hematoxylin.

Effect of Time Interval Between the Addition of Hematoxylin and Sodium Acetate

Since a faint red color is produced by the reaction of tin and hematoxylin in slightly acid solution, and requires up to 40 minutes for full development (8), it seemed advisable to find out whether this color had to be completely developed before adding the sodium acetate. Experiments showed that the same results were obtained whether the interval between the addition of the hematoxylin and sodium acetate was 10 seconds or 4 hours.

Effect of Time of Boiling

The effect of time of heating in the boiling water bath is shown in Figure 6. The color developed completely in 12 minutes and decreased when the heating time was more than 20 minutes. A 15 minute heating period is recommended. It is preferable to place the flasks into water that is boiling vigorously, and then turn down the flame so that the water boils moderately. When the water was boiled very vigorously for 15 minutes, slightly lower results were obtained. Only a very faint violet color developed when the solutions were not boiled. The boiling may have the effect of converting the hematoxylin to a degradation product (possibly hematin).

Effect of Temperature at Which the Color is Read

When the solutions were in the boiling water bath, they seemed to have more of an amber color than when cool. Therefore, the effect of temperature at which the color was read was investigated. The same readings were obtained over the range of 17° C. to 30° C.

Stability of the Color

The color was stable for 8 hours.

Effect of Valence of Tin

In the proposed method the tin is in the quadrivalent state, since it is oxidized by the treatment with nitric, perchloric, and sulfuric acids. To find out whether divalent tin would give the same result as quadrivalent tin, 3 ml of a standard tin solution (1 ml = 1.0 mg of Sn), freshly prepared by dissolving metallic tin in hydrochloric acid, was pipetted into a 250 ml volumetric flask containing water and 9.8 ml of sulfuric acid. The solution was diluted to the mark, a 5 ml aliquot taken, and the tin color developed as usual. The same result was obtained as with quadrivalent tin. The explanation may be that stannous tin is oxidized during the boiling. There is no danger of loss of tin on evaporating tin solutions containing hydrochloric acid to fumes of sulfuric acid, since neither stannous chloride (10) nor stannic chloride (4) are volatile under these circumstances.

Spectrophotometric Curves

The spectrophotometric curves obtained for the color and for the blank are shown in Figure 7. Water was used as the reference solution. There is a plateau for the transmittance of the color extending over the range of about 540 to 560 millimicrons, and maximum absorption probably occurs at about 550 millimicrons. The color of the blank increased over the length of the plateau region. From a consideration of Figure 7, it seemed most suitable to measure the transmittance at 590 millimicrons to reduce the effect of the blank.

Calibration Curve

A calibration curve was prepared as described under Preparation of Calibration Curve. It followed Beer's law.

Interferences

To test the possible effect of interfering metals 1 mg of the different metals were added to 5 ml of blank solution and the samples carried through the procedure. The findings are summarized in Table I. For elements that did not interfere, further tests were made in the presence of tin (Table I). Arsenic, barium, beryllium, boron, cadmium, calcium, cobalt, lead, lithium, magnesium, manganese, nickel, potassium, sodium, strontium, and zinc did not interfere. Aluminum, antimony, bismuth, chromium, copper, germanium, iron, molybdenum, titanium, thorium, tungsten, vanadium, and zirconium interfered by producing lakes. Cerium, mercury, selenium, and uranium interfered by giving colors of not too great intensity that were apparently not lakes. Silver interfered by producing a precipitate of silver chloride in the cold and elemental silver on boiling.

Lead, barium, and strontium did not precipitate as sulfates in the above tests because the solutions were mixed quickly after adding the corresponding salt (this was done purposely to be certain that no lakes would be produced by these elements). In the regular procedure barium, lead, and strontium sulfates precipitated after fuming with sulfuric acid and diluting. This was harmless, since in the presence of these metals (and also silica), the solutions were allowed to stand for 1 hour after diluting to the mark to permit the precipitate to settle.

The effect of anions was investigated by adding 0.1 gram of different salts to 50 ml volumetric flasks containing 0.060 mg of tin and then developing the colors. This amount of salt is equivalent to 5 grams in the original 250 ml volume. The results obtained are shown in Table II. Acetate, bromide, chlorate, chloride, nitrate, perchlorate, phosphate, sulfate, and tartrate did not interfere. Nitrite, sulfide, sulfite, and thiosulfate caused low results because of their reducing action, while chromate and iodate caused low results because of their oxidizing action. Fluoride, oxalate, and thiocyanate produced low results by forming complexes with the tin. Iodide interfered because iodine was liberated, and permanganate interfered because manganese dioxide was precipitated. Carbonate caused low results, probably because of the neutralization of the acid. Persulfate caused high results for some unknown reason. The interference of nitrite, sulfide, sulfite, thiosulfate, fluoride, oxalate, thiocyanate, carbonate, and persulfate would be eliminated by the fuming in the recommended method (a platinum dish would have to be used with fluoride). The non-interference of chloride, perchlorate, and phosphate would seem to indicate that the corresponding acids (or mixtures of these acids with sulfuric acid) might be used for adjusting the acidity before taking the aliquot. This was not investigated.

The technique employed in this paper will probably be useful for the colorimetric determination of other metals (especially aluminum, antimony, germanium, thorium, and zirconium) in addition to tin. It must be emphasized that the colors of the lakes shown in Table I were obtained with 1 mg of the elements. For smaller amounts, the colors would probably resemble the brown and amber colors obtained with tin. The violet color for the lake was produced when there was a very large excess of the metal over the dye. One milligram of tin also produced a violet lake.

**B. RECOMMENDED METHOD FOR THE DETERMINATION OF METALLIC
TIN IN PROPELLANTS**

Reagents

Hematoxylin solution (0.1%). Dissolve 0.1 gram of hematoxylin in 80 ml of 95% ethyl alcohol, add 1 drop of hydrochloric acid, and dilute to 100 ml with 95% ethyl alcohol. Prepare fresh daily.

Gelatin solution (1%). Dissolve 2 grams of gelatin in 200 ml of water by warming at about 50° C. Cool to room temperature. Prepare fresh every 2 days.

Sodium acetate solution (50%). Dissolve 500 grams of sodium acetate trihydrate in water and dilute to 1 liter.

Standard tin solution No. 1 (1 ml = 1.00 mg tin). Dissolve 1.0000 gram of pure granulated tin in 100 ml of hydrochloric acid by heating at 40° C. in a covered 400 ml beaker. Add about 300 ml of water, cool, and dilute to 1 liter in a volumetric flask.

Standard tin solution No. 2 (1 ml = 0.012 mg tin). Measure 3.0 ml of standard tin solution No. 1 into a 250 ml beaker, and add 10 ml of nitric acid, 10 ml of sulfuric acid, and 1 ml of perchloric acid. Cover with a watch glass, evaporate to fumes, and then with the cover lid ajar heat at the highest heat of the hot plate for 12 minutes. Cool, add water, cool again, and dilute to 250 ml in a volumetric flask.

Blank solution. Proceed as in the preparation of standard tin solution No. 2 but omit the tin.

Preparation of Calibration Curve

Measure 1.0, 2.0, 3.0, 4.0, and 5.0 ml of standard tin solution No. 1 into 250 ml beakers, and add 10 ml of nitric acid, 10 ml of sulfuric acid, and 1 ml of perchloric acid (70%). Carry along a reagent blank. Cover with watch glasses, evaporate to fumes, and then with the cover lids ajar heat at the highest heat of hot plate for 12 minutes. Cool, add water, cool again, and dilute to 250 ml in volumetric flasks.

Pipet 5 ml aliquots into 50 ml volumetric flasks, and add 15 ml of water, 4.0 ml hematoxylin solution (0.1%) 4 ml of gelatin solution (1%), and 10 ml of sodium acetate solution (50%). Measure the hematoxylin solution with a pipet or buret; measure the other reagents with a graduate. Place the volumetric flasks into 400 ml beakers containing 300 ml of tap water that is boiling vigorously, then turn down the heat so that the water boils moderately and keep the flasks in the water for 15 minutes.

Remove the flasks and cool to room temperature in a water bath. Dilute to the mark and measure the transmittance at 590 millimicrons with a spectrophotometer that has been set to 100 per cent transmittance with the reagent blank. Plot mg of tin in the 50 ml volumes against per cent transmittance.

Method

Use a sample of such size that the 5 ml aliquot from the 250 ml volume will contain preferably 0.05 to 0.08 mg of tin (the usual size sample will be 0.3 gram). Transfer the sample to a 250 ml beaker, and add 10 ml of nitric acid, 10 ml of sulfuric acid, and 1 ml of perchloric acid. Carry along a reagent blank. Cover with a watch glass, evaporate to fumes, and then with the cover lid ajar heat at the highest heat of the hot plate for 12 minutes. Cool, add water, cool again, and dilute to 250 ml in a volumetric flask. If barium, strontium, lead, or silica are present allow to stand for 1 hour at this stage (after shaking) so that the precipitate will settle. Pipet a 5 ml aliquot into a 50 ml volumetric flask, add 15 ml of water, and develop the color as described under the Preparation of Calibration Curve. Convert the reading to mg of tin by consulting the calibration curve. Calculate as follows:

$$\% \text{ Sn} = \frac{\text{Mg of Sn as read from curve}}{\text{Grams of sample in aliquot} \times 10}$$

Precaution: The volumetric flasks in which the color is developed and the spectrophotometer cells must be rinsed with hydrochloric acid before being used for the next determination.

C. RESULTS FOR TIN IN PROPELLANTS

The results obtained when a typical small arms propellant containing metallic tin was analyzed several times by the proposed method showed good accuracy and precision (Table III). Good recoveries were obtained on adding standard tin solution No. 1 to 0.3 gram portions of a propellant not containing tin and carrying the samples through the procedure (Table IV). Satisfactory results were obtained for synthetic samples containing lead stearate, barium nitrate, and strontium nitrate (Table IV).

Experiments were also carried out on a separation method, whereby the tin was first isolated as metastannic acid. This separation method offered no advantage over the direct method, but it did help establish the validity of the direct method since the same results were obtained.

In this separation method a 1.2 gram sample was treated with 35 ml of nitric acid and the solution boiled down to a volume of 10 ml. Fifty milliliters of water was added and the boiling continued for 5 minutes. The solution was immediately filtered through a fine filter paper and the precipitate washed with hot water. The precipitate and filter paper were treated with a mixture of 10 ml of sulfuric acid, 15 ml of nitric acid, and 3 ml of perchloric acid. The solution was fumed strongly for 15 minutes, and diluted to 1 liter after adding 30 more ml of sulfuric acid. A 5 ml aliquot was taken.

The above methods were not applicable to the determination of tin dioxide, since that compound was insoluble in fuming sulfuric acid. In an attempt to develop a "universal" method applicable to metallic tin and tin dioxide, much work was done on a fusion procedure whereby a 0.3 gram sample was treated with 5 ml of nitric acid in a platinum crucible, the solution evaporated to dryness, and the residue ignited for 3 minutes. The residue was then fused with 8 grams of potassium bisulfate, and 10 ml of sulfuric acid was added 25 seconds after turning off the flame (in this way no salts precipitated). The solution was then diluted to 250 ml and a 5 ml aliquot taken. Although the method seemed attractive in principle, the precision was poor ($\pm 0.2\%$), probably because of the potassium bisulfate and the platinum picked up during the fusion. The method may be useful for determining tin dioxide in propellants when only a small amount of sample is available (the gravimetric method is not satisfactory for small size samples). In applying the fusion method it is essential that the platinum crucible be thoroughly cleaned by a bisulfate fusion to remove traces of iron.

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VI. TABLES

Table I. Effect of 1 Mg of Diverse Metals on the Colorimetric Determination of Tin

<u>Element</u>	<u>Added as</u>	<u>Interference in Absence of Tin</u>	<u>Interference in the Presence of 0.060 Mg Tin (Tin Recovered, Mg)</u>
Aluminum	AlCl ₃	Violet lake	---
Antimony	SbCl ₃	Violet lake	---
Bismuth	BiONO ₃	Violet lake	---
Chromium	Cr(NO ₃) ₃	Blue-violet lake	---
Copper	CuSO ₄	Red-violet lake	---
Germanium	GeO ₂	Violet lake	---
Iron	Fe ₂ (SO ₄) ₃	Violet lake	---
Molybdenum	(NH ₄) ₂ MoO ₄	Blue-violet lake	---
Thorium	Th(NO ₃) ₄	Red-violet lake	---
Titanium	TiCl ₄	Violet lake	---
Tungsten	Na ₂ WO ₄	Violet lake	---
Vanadium	(VO) ₂ Cl	Red-violet lake	---
Zirconium	Zr(NO ₃) ₄	Violet lake	---
Cerium	Ca(SO ₄) ₂	Orange color	---
Mercury	HgCl ₂	Turbid brown color	---
Selenium	H ₂ SeO ₃	Brown color	---
Uranium	UO ₂ (C ₂ H ₃ O ₂) ₂	Orange color	---
Silver	AgNO ₃	Silver chloride and elemental silver precipitated	---
Arsenic	As ₂ O ₅	None	0.060
Barium	BaCl ₂	None	0.058
Beryllium	BeSO ₄	None	0.060
Boron	H ₃ BO ₃	None	0.059
Cadmium	CdCl ₂	None	0.059
Calcium	CaCl ₂	None	0.062
Cobalt	CoCl ₂	None	0.058
Lead	Pb(C ₂ H ₃ O ₂) ₂	None	0.061
Lithium	LiCl	None	0.059
Magnesium	MgSO ₄	None	0.060
Manganese	MnSO ₄	None	0.062
Nickel	NiCl ₂	None	0.059
Potassium	K ₂ SO ₄	None	0.061
Sodium	Na ₂ SO ₄	None	0.058
Strontium	SrCl ₂	None	0.058
Zinc	ZnCl ₂	None	0.060

Table II. Results for Tin in the Presence of Anions (0.060 Mg Tin Present)

<u>Anion</u>	<u>Salt Added (0.1 Gram)^a</u>	<u>Tin Found (Mg)</u>
Acetate	NaC ₂ H ₃ O ₂	0.062
Bromide	NaBr	0.058
Chlorate	NaClO ₃	0.059
Chloride	NaCl	0.060
Nitrate	NaNO ₃	0.058
Perchlorate	NH ₄ ClO ₄	0.062
Phosphate	Na ₂ HPO ₄	0.058
Sulfate	Na ₂ SO ₄	0.060
Tartrate	(NH ₄) ₂ C ₄ H ₄ O ₆	0.063
Nitrite	NaNO ₂	0.023
Sulfide	Na ₂ S	0.00
Sulfite	Na ₂ SO ₃	0.00
Thiosulfate	Na ₂ S ₂ O ₃	0.00 ^b
Chromate	Na ₂ Cr ₂ O ₇	0.036
Iodate	NaIO ₃	0.018
Fluoride	KF	0.036
Oxalate	Na ₂ C ₂ O ₄	0.034
Thiocyanate	NaCNS	0.030
Iodide	KI	0.071
Permanganate	KMnO ₄	Manganese dioxide precipitated
Carbonate	Na ₂ CO ₃	0.038
Persulfate	(NH ₄) ₂ S ₂ O ₈	0.074

^a Water of crystallization not included.

^b Visual estimation because some sulfur precipitated.

Table III. Results for Tin in a Typical Propellant Containing Tin^a

	<u>Tin Found (%)</u>
	0.98
	0.95
	1.00
	0.95
	0.95
	0.98
	1.00
Ave.	0.97
Std. Dev.	0.023

^a Contained 0.95% tin as determined iodometrically (6), 89.02% nitro-cellulose, 0.65% diphenylamine, 0.94% potassium sulfate, 6.71% dinitrotoluene, 0.90% moisture.

Table IV. Recovery of Tin from Synthetic Samples

<u>Synthetic Sample</u>	<u>Tin Found (Mg)</u>
0.3 gram M-9 propellant ^a + 0.00 mg tin	0.00
0.3 gram M-9 propellant + 0.50 mg tin	0.45
0.3 gram M-9 propellant + 1.00 mg tin	1.00
0.3 gram M-9 propellant + 2.00 mg tin	2.05
0.3 gram M-9 propellant + 3.00 mg tin	3.05
0.3 gram M-9 propellant + 4.00 mg tin	4.05
0.3 gram M-9 propellant + 5.00 mg tin	4.90
0.3 gram M-9 propellant + 3.00 mg tin + 0.03 gram lead stearate	3.05
0.3 gram M-9 propellant + 3.00 mg tin + 0.015 gram barium nitrate	3.05
0.3 gram M-9 propellant + 3.00 mg tin + 0.015 gram strontium nitrate	2.95

^a Contained 57.75% nitrocellulose, 40.0% nitroglycerin, 1.5% potassium nitrate, 0.75% diphenylamine.

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March 1961

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- 1 - Commanding Officer
Longhorn Ordnance Works
Marshall, Texas
ATTN: Mr. M. R. Kesling
- 2 - Stanford Research Institute
Roulter Laboratories
Menlo Park, California
ATTN: Dr. R. F. Muraca
Dr. Eugene Burns
- 1 - Commanding Officer
U. S. Naval Ammunition and
Net Depot
Seal Beach, California
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| 1 - Commanding Officer
Office of Ordnance Research
Box CM, Duke Station
Durham, North Carolina | 1 - U. S. Atomic Energy Commission
Division of Military Application
Washington 25, D. C. |

10 - Commander
Armed Services Technical Info Agcy
Arlington Hall Station
Arlington 12, Virginia
ATTN: TIPCR

1 - Solid Propellant Info Agcy
Applied Physics Laboratory
The Johns Hopkins University
8621 Georgia Avenue
Silver Spring, Maryland

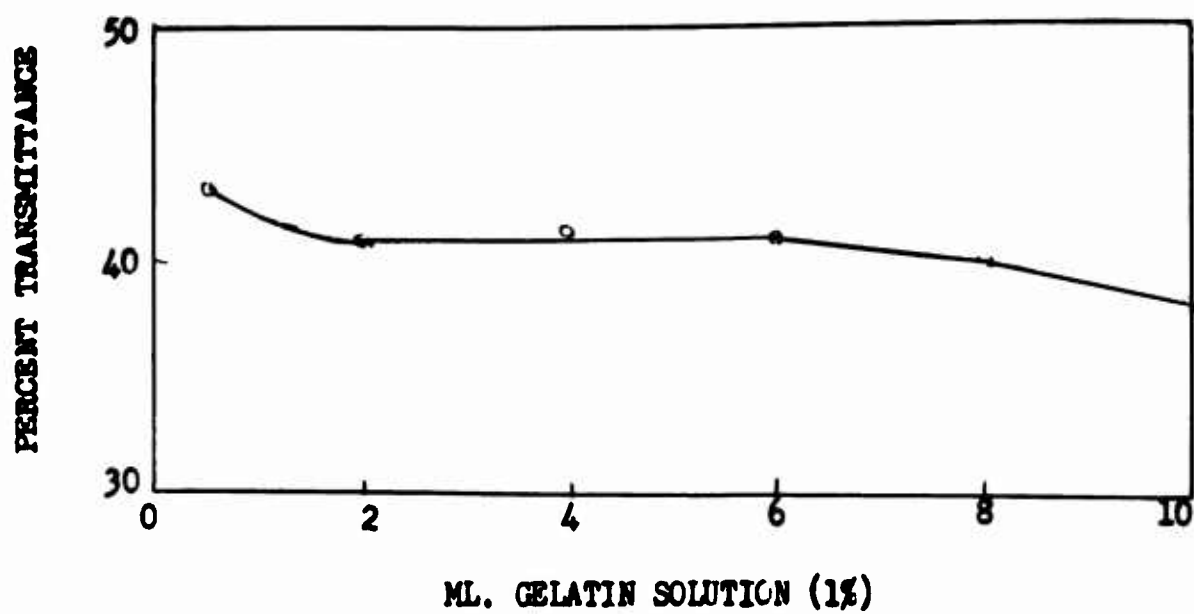


Figure 1. Effect of amount of gelatin on color

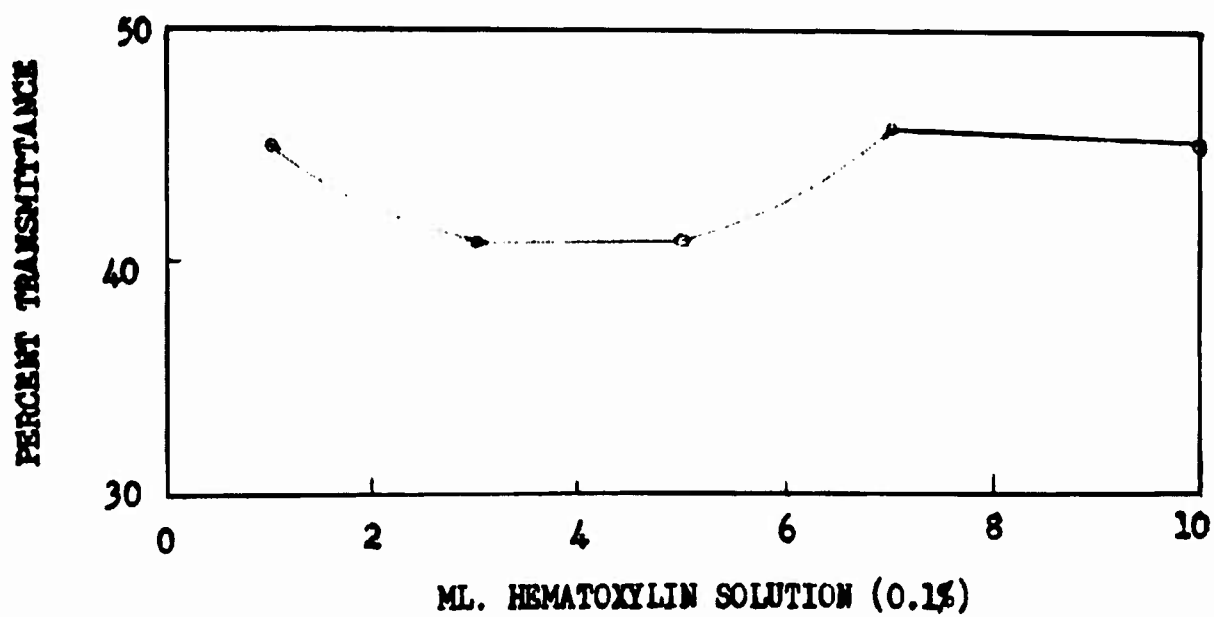


Figure 2. Effect of amount of hematoxylin on color

88-231.5 1523-080-59.

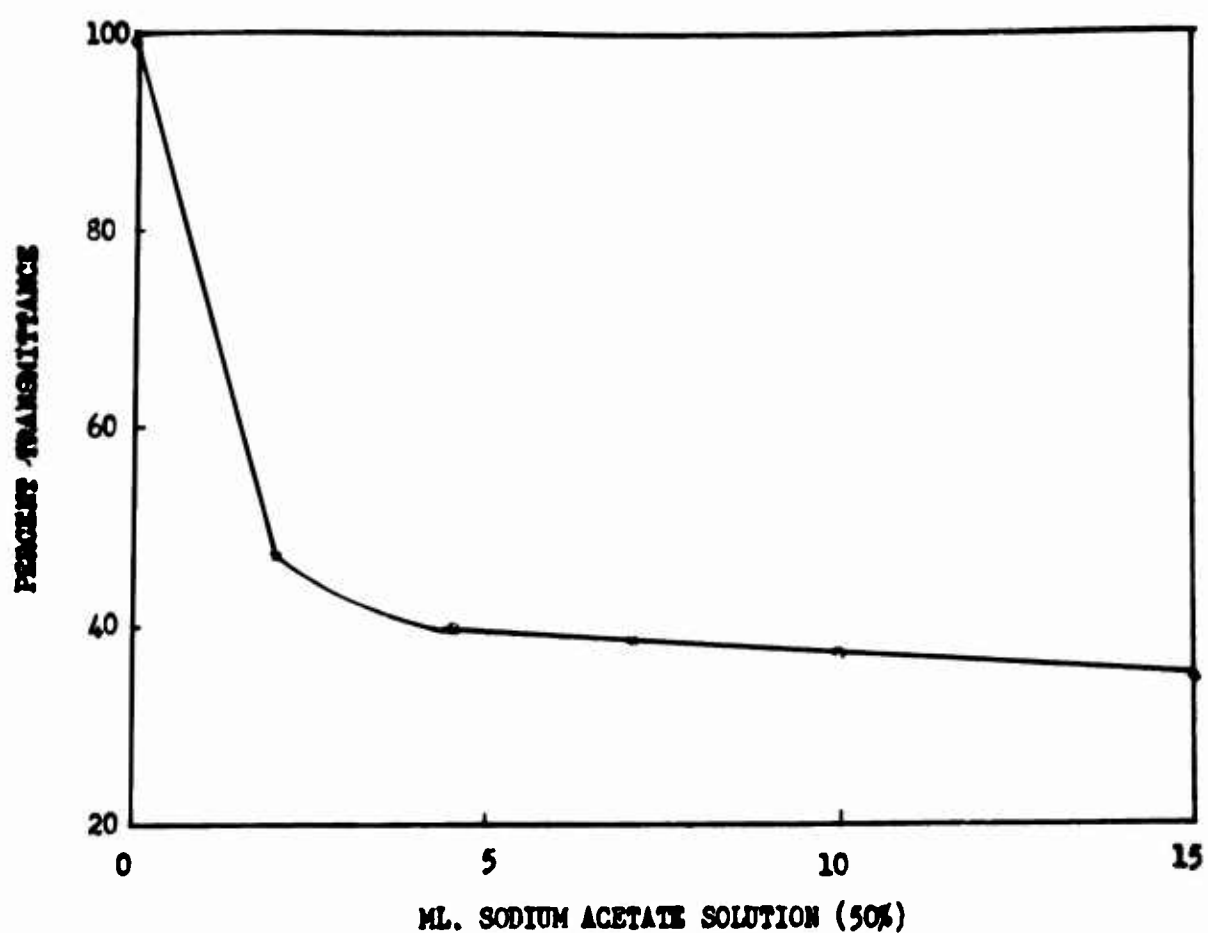


Figure 3. Effect of amount of sodium acetate on color

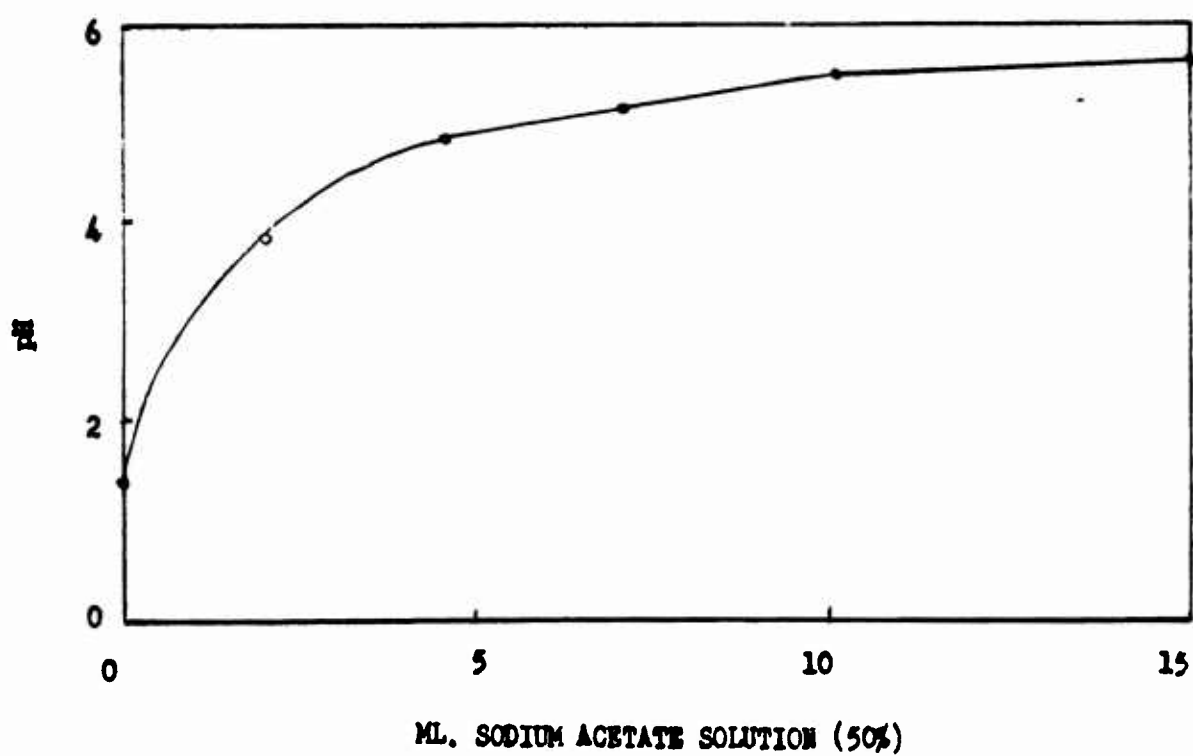


Figure 4. Effect of amount of sodium acetate on pH

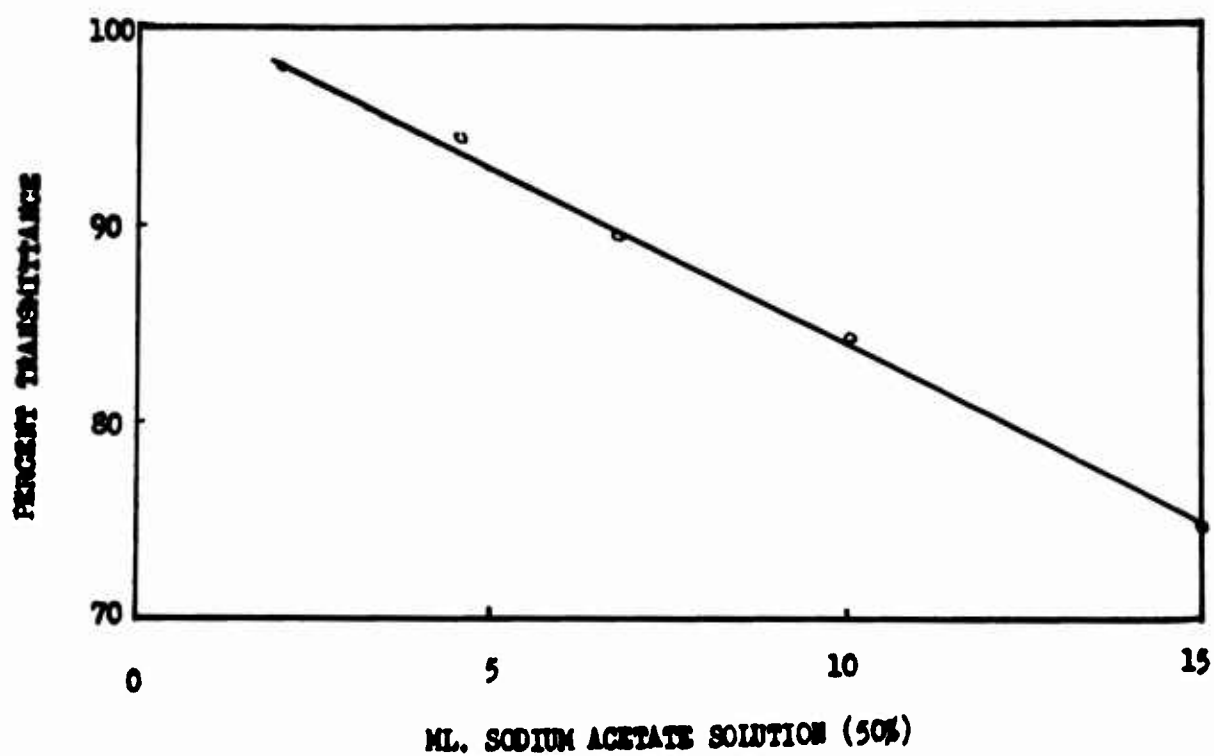


Figure 5. Effect of amount of sodium acetate on blank

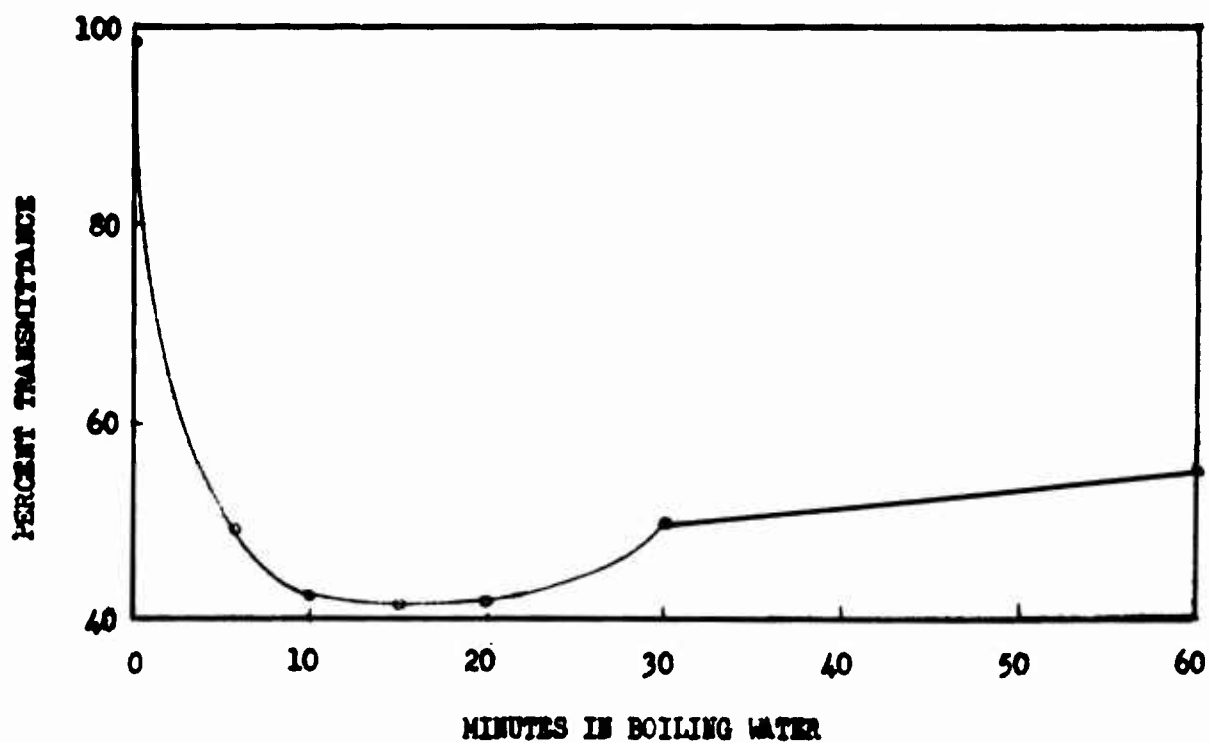


Figure 6. Effect of time of boiling on color

78.031.3 15.25.080.59.

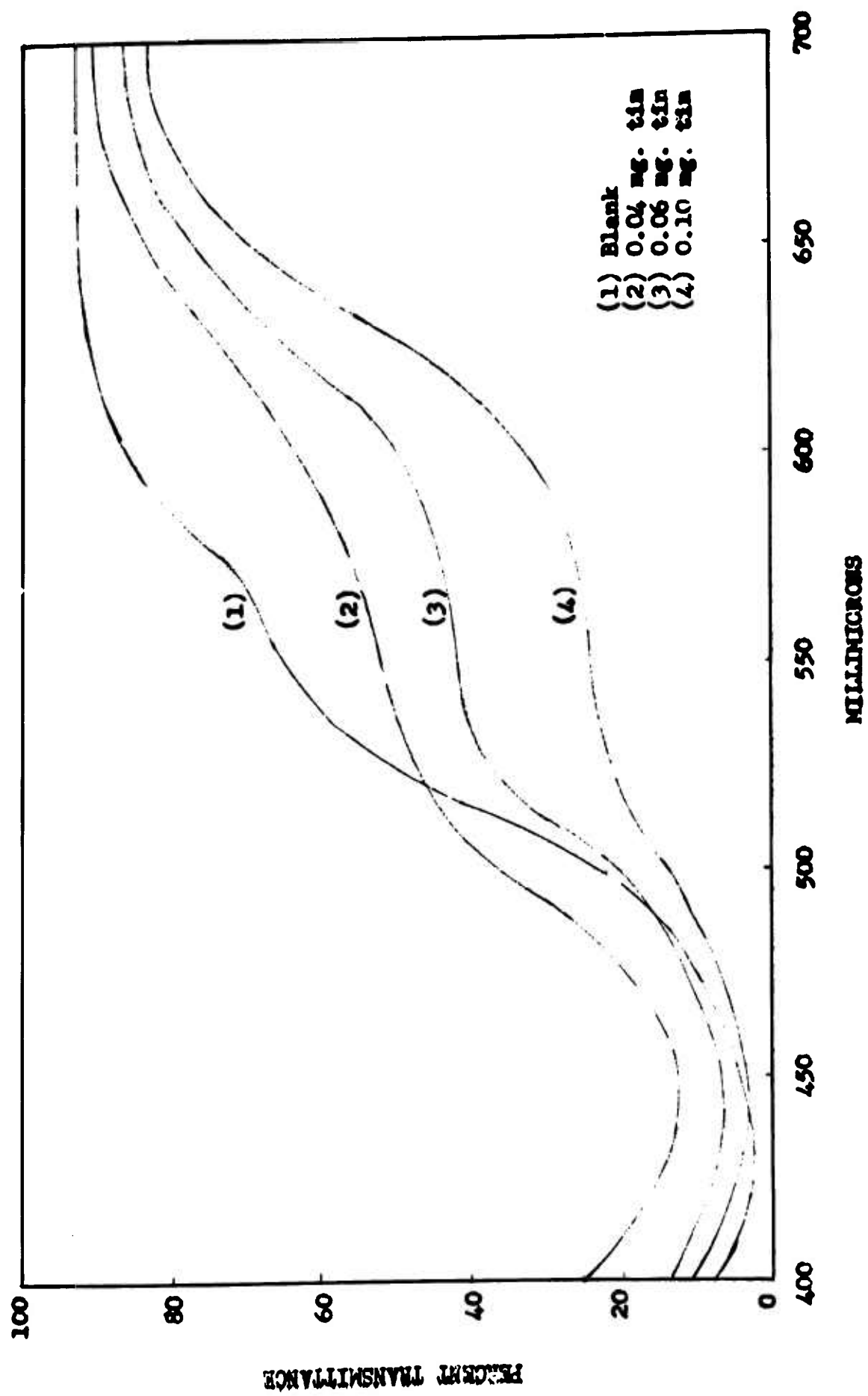


Figure 7. Spectrophotometric curves for color and blank

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